Recycling of residual IGCC slags and their benefits as degreasers in ceramics

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ABSTRACT

This work studies the evolution of IGCC slag grains within a ceramic matrix fired at different temperatures to investigate the effect of using IGCC slag as a degreaser. Pressed ceramic specimens from two clay mixtures are used in this study. The M1 mixture is composed of standard clays, whereas the M2 mixture is composed of the same clay mixture as M1 mixture but contains 15% by weight IGCC slag. The amount of IGCC slag added coincides with the amount of slag typically used as a degreaser in the ceramic industry. Specimens are fired at 950 °C, 1000 °C, 1050 °C, 1100 °C and 1150 °C. The mineralogical composition and the IGCC slag grain shape within the ceramic matrix are determined by X-ray diffraction, polarized light microscopy and scanning electron microscopy. The results reveal that the surface of the slag grains is welded to the ceramic matrix while the quartz grains are separated, which causes increased water absorption and reduces the mechanical strength. IGCC slag, however, reduces water absorption. This behaviour is due to the softening temperature of the slag. This property is quite important from an industrial viewpoint because IGCC slag can serve as an alternative to traditional degreasing agents in the ceramic building industry. Additionally, using IGCC slag allows for the transformation of waste into a secondary raw material, thereby avoiding disposal at landfills; moreover, these industrial wastes are made inert and improve the properties of ceramics.

Keywords: IGCC slag, Scanning electron microscopy Anorthite, Devitrification Thermal, b e haviour, Ceramic petrography, Degreaser

1. Introduction

Thermal Power Integrated Gasification Combined Cycle (IGCC) plants produce electricity from the combustion of synthesis gas in a combined cycle process. This gas is obtained from a solid or liquid fuel in a gasification process in which residual IGCC slags are produced. IGCC technology was largely developed for environmental reasons; thus, it is important to study the waste materials generated in order to find applications and reduce slag waste. Currently, there is much concern about the increasing amount of industrial waste. The ceramic process allows for recycling of different waste materials, such as glass, biodiesel ashes, steel slag, organic residues, sugarcane bagasse, biomass ashes, spent foundry sands, PC and RV waste glass and fly ashes and by-products of different industrial process (Barba et al., 2002; Shih et al., 2004; Menezes et al., 2005; Aineto et al., 2006a,b; Demir, 2008; Dondi et al., 2009; Iglesias et al., 2009; Loryuenyong et al., 2009; Alonso-Santurde et al., 2011; Karamanova et al., 2011; Faria et al., 2012; He et al., 2012; Pérez-Villarejo et al., 2012). Recycling IGCC slag has both environmental and economic benefits and also improves the technological properties of ceramics (Acosta et al., 2002b).

In Puertollano (Ciudad Real, Spain), there is an IGCC power plant that uses coal as fuel as well as petroleum coke. 80.000 tons of IGCC slags are produced in this plant each year (Treviño, 2003; Anónimus, 2001). Acosta et al. (2001), Acosta et al. (2002a,b), and Aineto et al. (2006a,b) have characterized this slag waste and studied its behaviour as a degreasing material for pressed bricks that are fired at approximately 950 °C.

IGCC slag is a solid, glassy, granular and black material that is 70% composed of silica and alumina, plus oxides of calcium and iron (Acosta et al., 2001). It is a "short glass" that, when heated from 800 °C to 1050 °C, undergoes a devitrification process and is transformed into different minerals (hematite, anorthite and cristobalite) (Acosta et al., 2002a). It can be used as degreasing in ceramic materials because of its physical and chemical properties. Acosta et al. (2002b) have studied the behaviour of ceramic bricks with varying amounts of IGCC slag and concluded that this slag works well as a degreaser.

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Slag reduces the plasticity and the linear shrinkage of both raw and dry paste in the same way as other degreasing materials, such as quartz and carbonate sands. Degreasing materials reduce the mechanical strength and increase water absorption, whereas slag improves these properties (reduces water absorption and increases mechanical strength). The results of these investigations have been proven on an industrial scale: soft-paste bricks containing 15% slag from the IGCC plant in Puertollano (Ciudad Real) have been manufactured and commercialized.

Not much research has been performed on this topic because using slag as a degreasing material is a recent technology. However, the fact that IGCC slag improves water absorption and the mechanical strength of materials makes it quite interesting for industrial applications because traditional degreasing materials do not display such properties. The research conducted in this work can open new avenues of study for this material, which can be applied not only in Spain but also in other countries that have developed this technology. Two of the IGCC Power Plants are in the U.S. and two in Europe (Puertollano, Spain and Bughenum, Holland). Consequently, there are few studies on IGCC slags, and the research presented here is novel.

This work investigates the causes of the reduction in water absorption and the increase in mechanical strength in ceramic samples containing slag with the aims to understand the thermal behaviour of slag.

2. Materials and methods

The materials used in this research are a mixture of industrial clays and IGCC slags. The clays come from "Rústicos La Mancha, S.L.", Santa Cruz de Mudela (Ciudad Real, Spain) and are used for the production of soft-paste bricks. These clays date back to the Miocene era. They are highly clayed samples, composed of quartz, calcite, dolomite, illite, kaolinite, smectite and minor amounts of palygorskite. These clays have a very fine grain size, appropriate for pottery making. However, they are not useful for brick making due to their high plasticity and very high carbonate content (up to 50% of weight). For brick making, it is necessary to mix the clay with degreasing materials, such as quartz sands. The slag comes from the Elcogas, S.A. Power Station IGCC Puertollano (Ciudad Real, Spain). IGCC slag is a granular glassy, black material. Its chemical composition is silica and alumina, which comprise more than 70% of its total weight. Other important oxides are CaO and Fe_2O_3. Loss of ignition is less than 2% (Acosta et al., 2001). IGCC slag, when heated, produces hematite and devitrificates into anorthite and cristobalite (Acosta et al., 2002a). It is a short glass with a melting point above 1300 °C. The chemical compositions of the clays and IGCC slags have been determined by X-ray fluorescence.

This study was conducted on ceramic specimens made of two different mixtures: M1 and M2. Both are composed of Santa Cruz de Mudela clays; M2 contains 15% IGCC slag to observe the effect of slag on the fired ceramics. The amount of slag chosen coincides with the amount of slag used as degreaser in the ceramic factory "Rústicos La Mancha, S.L." of Santa Cruz de Mudela. The amount of degreaser depends on each clay mixture (plasticity and chemical composition). In this work, these factors we have been considered. The addition of 15% of slag in M2 mixture produces normal values of plasticity and chemical composition for common clays used on tiles and bricks (Facincani, 1992). Slag particles were milled to less than 0.5 mm in size. 50 cylindrical specimens were prepared by pressing, with a pressure of 35 MPa. The specimens are cylindrical, 4 cm in diameter, and 1 cm thick and were dried for 24 h at 80 °C. They were fired at 950 °C, 1000 °C, 1050 °C, 1100 °C and 1150 °C; the last temperature was held for 2 h. To determine the ceramic properties of the fired specimens, the diametral shrinkage, the water absorption (UNE, 1998) and the mechanical strength were measured using the Brazilian test (Iglesias et al., 2011).

The mineralogy of raw materials as well as the properties of ceramics fired at different temperatures were studied to determine the effect of IGCC slag. Mineralogical characterization was performed by X-ray diffraction (XRD) using a Philips X 'Pert MPD diffractometer with Cu Ka radiation and a graphite monochromator. Bulk mineralogy was studied using random-powder specimens, scanned from 2 to 75° at a scan speed of 0.02° 2q/s. Interpretation of the diffractograms was performed using files from the ASTM (American Society for Testing and Materials) compiled by the JCPDS (Joint Committee on Powder Diffraction Standards).

The ceramic specimens have been studied with polarized light microscopy and scanning electronic microscopy to determine the relationship between the IGCC slag grains and the ceramic matrix. Polarized light microscopy was performed for petrographic studies. Thin sections from the interior of the specimens, parallel to its height, were studied with both transmitted and reflected light with 10x eyepiece objectives at 2.5x, 10x and 25x, with both cross and parallel nicols. The same sections were observed by scanning electron microscopy (SEM-FEG) to study the textural and morphological relationship between the ceramic matrix and the slag grains following the firing process. SEM-FEG were performed on representative samples of broken surfaces coated by gold, using a JSM-6330F Field Emission Scanning Electron Microscope, operating at 10 kV and W15 mm.

3. Results and discussion

The mineralogical composition of the clay mixtures is about 50% phyllosilicates (illite, 35%, kaolinite, 10% and smectite <5%), quartz (30%), dolomite (15%), calcite (<5%), and less than 3% feldspar, hematite and rutile. The chemical composition of both mixtures (M1, M2) and the slag sample are listed in Table 1. There are no relevant changes in the chemical composition as a consequence of the addition of slag to the clay samples because the chemical compositions of both are similar.

For ceramic materials, both mixtures are well formulated, and their ceramic properties evolve with temperature as expected, as

Table 1
Chemical analysis (XRF) of materials. SL: IGCC Slag; M1: clay mixture without IGCC slag; M2: clay mixture with 15% IGCC slag; LOI: Loss On Ignition at 1050 °C.

Elements (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	TiO_2
SL	52.37	19.68	6.61	12.33	1.48	2.09	0.29	2.03	0.64
M1	43.651	19.635	9.393	5.017	3.814	2.83	0.742	0.074	0.779
M2	44.167	19.981	8.897	5.797	4.153	2.945	0.777	0.186	0.809
Elements (%)	MnO	SrO	NiO	ZrO_2	BaO	P_2O_5	Cr_2O_3	CuO	LOI
SL	0.07	0.02	0.05	0.02	0.07	0.06	0.04	0.01	1.64
M1	0.072	0.031	0.001	0.016	0.077	0.093	0.016	0.012	13.67
M2	0.081	0.032	0.011	0.016	0.076	0.102	0.022	0.012	11.914

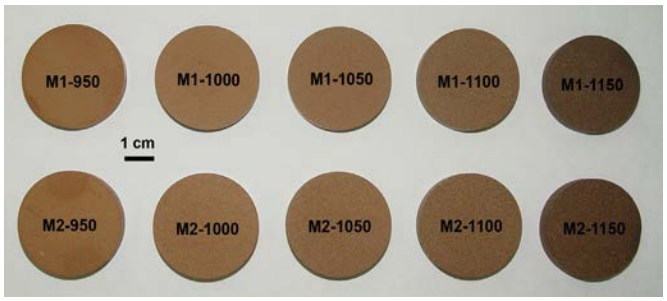

Fig. 1. M1 and M2 ceramic specimens. In the upper row M1, in the lower row M2. Fired temperatures from the left to the right: 950 °C, 1000 °C, 1050 °C, 1100 °C and 1150 °C.

evidenced by the results of the technological tests (Fig. 2) performed on the ceramic specimens fired at 950 °C, 1000 °C, 1050 °C, 1100 °C and 1150 °C. The micrographs in Fig. 1 show that the colour and surface morphology of both mixtures is typical for ceramic materials with these compositions fired at these temperatures. The changes in colour as a function of temperature range from orange to dark brown. No efflorescence was detected in any of the specimens. Slag grains appear as black dots in the specimens fired at 1050 °C; above this temperature they acquire a brown colour and a somewhat rough-textured surface.

Fig. 2 shows the results of the diametral shrinkage, water absorption and tensile strength. The amount of diametral shrinkage increases from 0.3% at 950 °C to 5.4% at 1150° C in M1 mixture while in the M2 mixture, the amount of shrinkage increases from 0.2% at 950 °C to 3.5% at 1150 °C. The amount of water absorption decreases from 21.06% at 950 °C to 6.11% at 1150 °C in the M1 mixture, while in the M2 mixture, water absorption decreases from 18.54% at 950 °C to 6.64% at 1150 °C. Both mixtures display a turning point at 1100 °C (Fig. 2a), which coincides with the change in colour and in external appearance observed in specimens above 1100 °C (Fig. 1); thus, 1100 °C is the sintering temperature of the material. The amounts of diametral shrinkage and water absorption are lower in M2 mixture than in M1 mixture; therefore, addition of IGCC slag improves the properties of the ceramic mixture. As shown in Fig. 2b the mechanical strength increases from 5.22 MPa

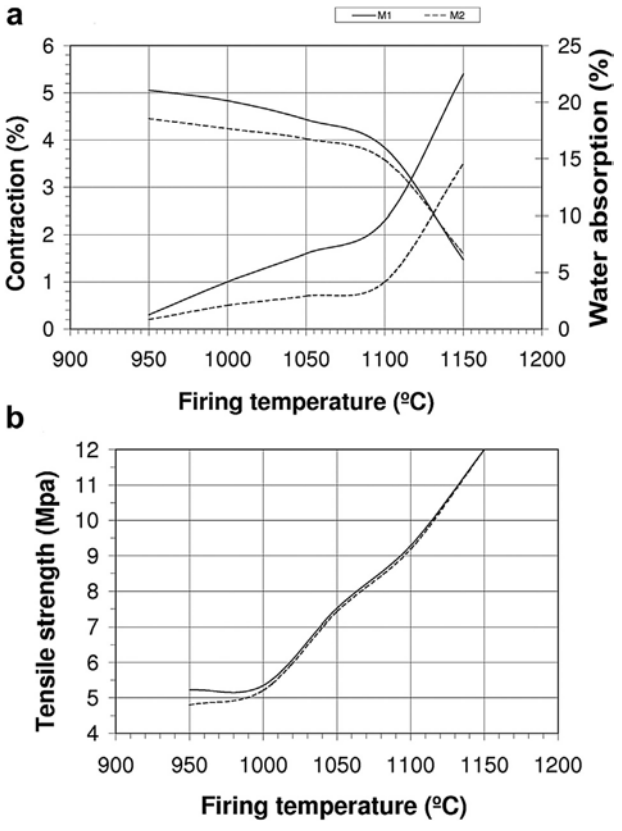

Fig. 2. Average results of the technological tests. a. Contraction and water absorption; b. Tensile strength.

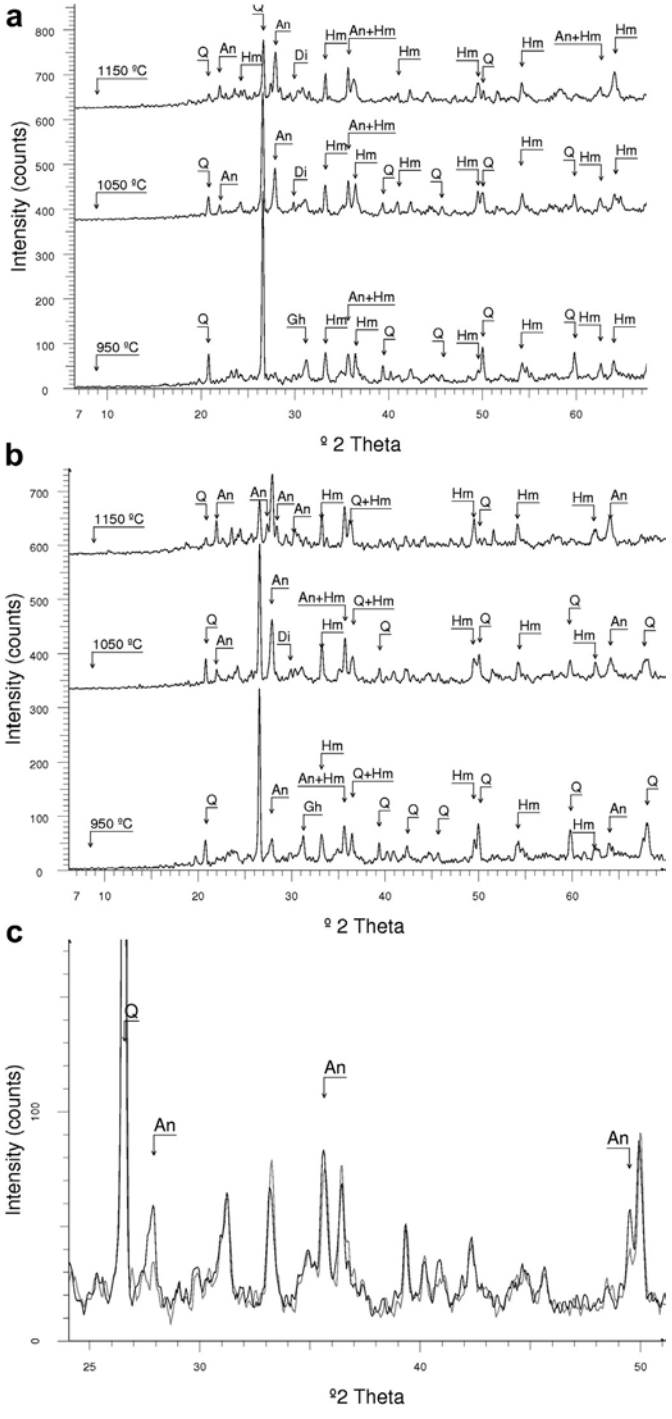

Fig. 3. XRD fired specimens. a. M1 mixture. b. M2 mixture. c. M1 and M2 mixtures at 950 °C. Red: M1 mixture; Black: M2 mixture. An: anorthite. Q: quartz; Di: diopside; Gh: gehlenite; Hm: hematite.

at 950 °C to 12 MPa at 1150 °C in M1 mixture and from 4.80 MPa at 950 °C to 12 MPa at 1150 °C in M2 mixture, these values are similar in both mixtures. All measured values, for both mixtures, lie within the normal interval of values for common clays used in the manufacture of tiles and bricks (Barba et al., 2002). Their evolution as a function of temperature is considered normal. The IGCC slag addition reduces the water absorption until 1100 °C; from this temperature the water absorption of M1 mixtures is the same that M2 mixture due to the sintering of the ceramic materials. A turning point occurs about 1100 °C as a consequence of the higher densification of the ceramic. The IGCC slag addition improves the diametral shrinkage for all the temperature range whereas the tensile strength does not decreases as occurs with the addition of other degreasing materials.

Fig. 3 shows the diffractograms of M1 and M2 mixtures, firing at 950 °C, 1050 °C and 1150 °C. The mineral phases identified in firing M1 mixture (Fig. 3a) were quartz, gehlenite, hematite, anorthite and diopside. Quartz is a mineral inherited from the raw material, whereas the other minerals identified are neoformed minerals that appear as a consequence of firing the raw material. The appearance of diopside is due to dolomite in raw materials. As the temperature increases above 1050 °C, gehlenite disappears, scarce mullite appears, the relative amounts of both anorthite and hematite increase while the amount of quartz decreases. The glass phase undergoes a progressive gain with increasing temperature. The formation of these minerals, as well as their evolution, is expected based on the chemical composition of the starting clay material. The appearance of anorthite at 950 °C is consistent with the results of previous studies by other authors (Barba et al., 2002) in which this mineral was formed in samples containing carbonates at this temperature. The minerals found in M2 mixture (Fig. 3b) are the same as those in M1 mixture. The development of these minerals as a function of temperature is also the same. However, there is a remarkable increase in the reflections of anorthite in M2 mixture (Fig. 3c). This anorthite corresponds to devitrification of the slag caused by the gradual warming that occurs during the firing process. The projection of the major chemical elements of IGCC slag (SiO_2, Al_2O_3 and CaO as shown in Table 1) in a ternary phase diagram SieAleCa shows that the slag plots in the anorthite stability field (Acosta et al., 2001; Acosta et al., 2002a, 2002b). Furthermore, both mixtures evolve in the same way, indicating that the slag devitrifies without affecting the formation of the mineral phases in the ceramic matrix.

Polarized light microscopy reveals that in both mixtures (M1 and M2), the evolution of the ceramic matrix after the firing process is similar. The colour changes from orange to dark brown due to the higher degree of vitrification (Fig. 4). The ceramic matrix has a very fine grain. In both M1 and M2 mixtures, the degreaser materials grains, such as quartz and feldspar, stand out. In addition, IGCC slag grains stand out in the M2 mixture. These grains are distributed evenly in both mixtures.

Anorthite crystals have been observed inside the IGCC slag grains after the firing process due to devitrification. Fig. 4 shows micrographs of the IGCC slag grains fired at 950 °C and 1150 °C; the colour and degree of devitrification are visible. Fig. 4aec show the same thin section of M2 mixture fired at 950 °C. The micrographs

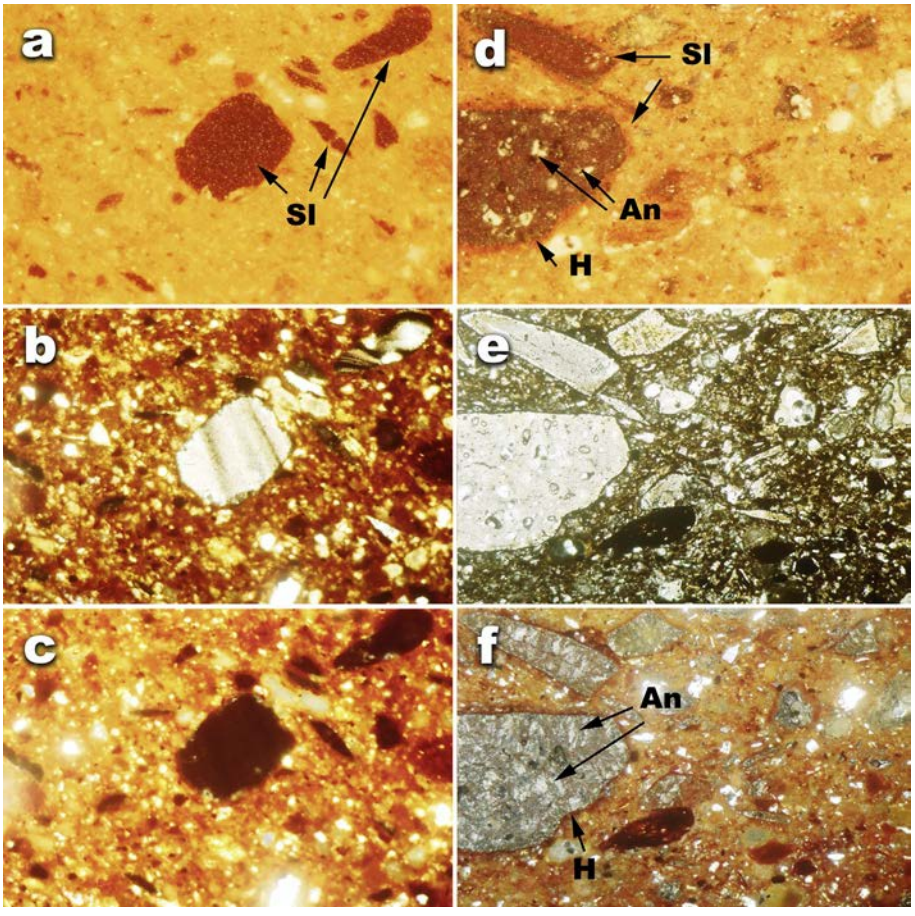

Fig. 4. Petrographic micrographs of M2 fired specimens. Left column: 950 °C, a: reflected light; b: transmitted light and parallel nicols; c: transmitted light and crossed nicols. Right column: 1150 °C, d: reflected light; e: transmitted light and parallel nicols; f: transmitted light and crossed nicols. SL: IGCC Slag; An: anorthite; H: halo.

show slag grains viewed under reflected light (Fig. 4a), transmitted light (Fig. 4b) and crossed nicols (Fig. 4c). IGCC slag grains maintain their black colour at 950 °C when seen through reflected light (Fig. 4a). However, when seen with transmitted light they are translucent (Fig. 4b) and with crossed nicols, they are black because they remain vitreous at this temperature. Hematite crystals are seen as a mass of small red dots, which leads to a reddish colour in the ceramic matrix (Fig. 4b). Devitrification of the slag begins at 850 °C (Acosta et al., 2002a); however, at 950 °C, most of the grains retain their original shape and have sharp edges, with no evidence of crystallization. The slag grains are joined to the ceramic matrix.

As the temperature increases gradually, the slag grains undergo devitrification without losing their external morphology. Fig. 4def shows the same thin section of M2 mixture fired at 1150 °C, for grains of IGCC slag viewed under reflected light (Fig. 4d), transmitted light (Fig. 4e) and crossed nicols (Fig. 4f). Under reflected light at 1150 °C, the IGCC slag grains possess a brown colour with white inclusions corresponding to anorthite crystals, which are formed during the firing process (Fig. 4d). Under transmitted light, the slag grains are translucent (Fig. 4e), whereas under crossed nicols, the slag grains are brown in colour and have inclusions of clear interference colours corresponding to small crystals of anorthite and cristobalite (Fig. 4f). The brown colour is caused by devitrification of glassy IGCC slag, which forms hematite (Acosta et al., 2002a). The white and grey crystals are assumed to correspond to anorthite and cristobalite because of their interference colours and because these are the minerals that appear during the devitrification of IGCC slag (Acosta et al., 2002a). The slag grains preserve their angular external morphology because they do not melt. The halo around the slag grains seems to be the result of the transformation of iron into hematite. The edges of the slag grains and the ceramic matrix are joined. Pores between the slag grains and the ceramic matrix are not observed.

Scanning electron microscopy (Fig. 5) shows that the ceramic matrix both M1 and M2 samples coincides at every temperature. This is corroborated by polarized light microscopy. At low temperatures, the matrix in both mixtures has a texture characteristic of these materials when fired at temperatures below 1050 °C. There is interparticle porosity that shows no signs of incipient fusion. At 950 °C the shape of clay mineral particles without sintering can be distinguished (Fig. 5a and b). Microgranular aggregation appears between the gaps of the ceramic matrix, which corresponds to the

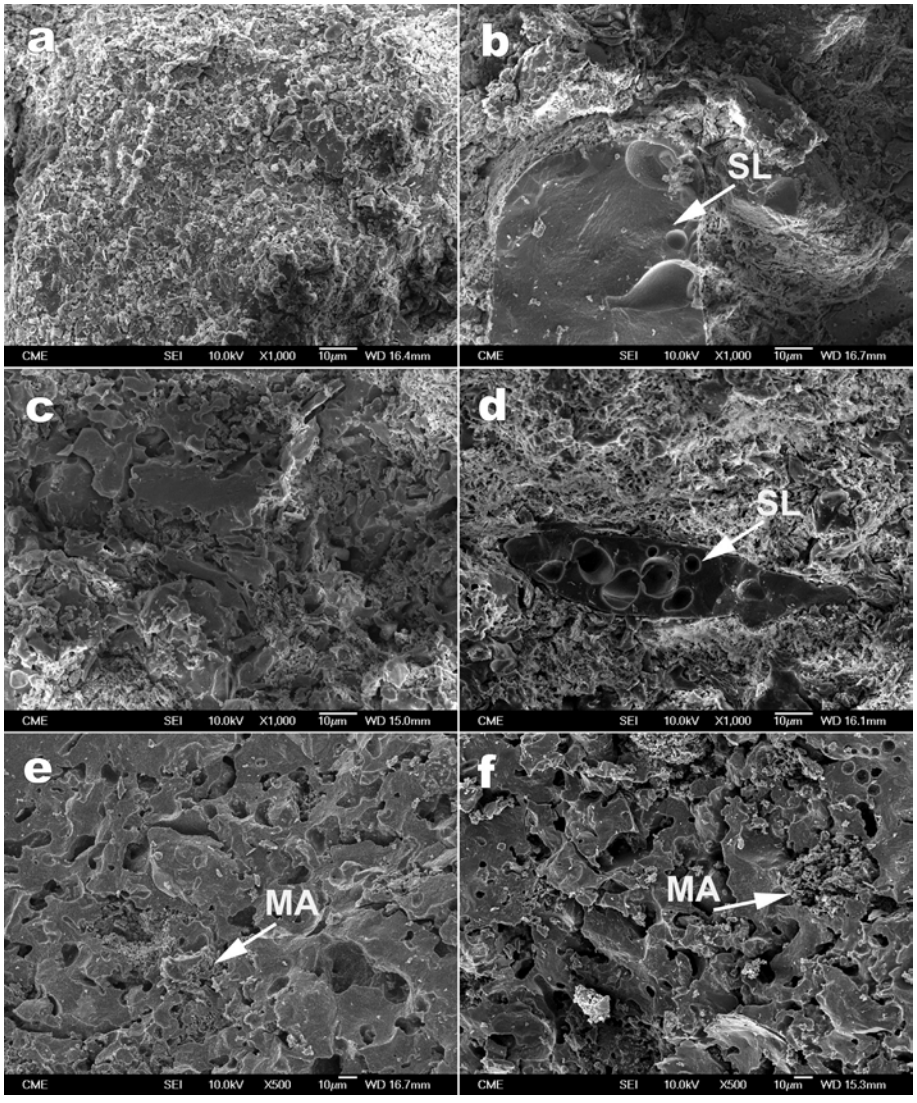

Fig. 5. SEM-FEG micrographs of M1 and M2 ceramic specimens. a: M1 ceramic matrix at 950 °C. b: M2 ceramic matrix and slag grain at 950 °C. c: M1 ceramic matrix at 1050 °C. d: M2 ceramic matrix and slag grain at 1050 °C. e: M1 ceramic matrix at 1150 °C. f: M2 ceramic matrix and slag grain at 1150 °C. SL: IGCC slag grain. MA: Microgranular aggregates.

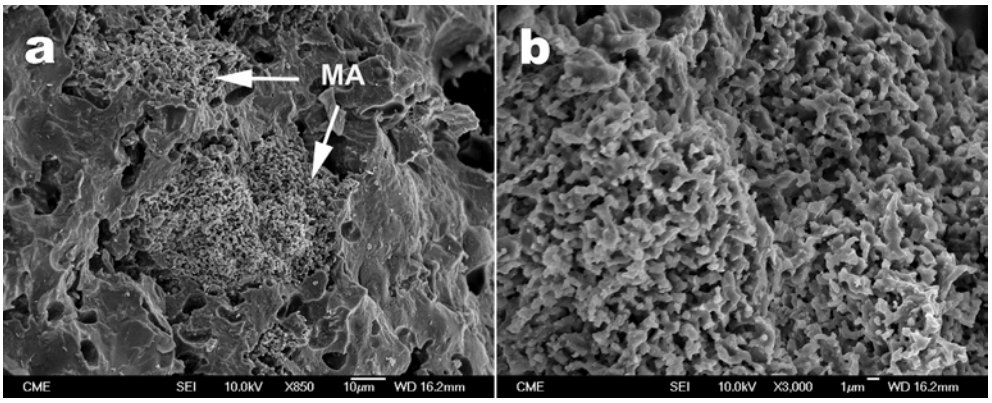

Fig. 6. SEM-FEG micrographs. a: calcium aluminosilicate microgranular aggregates in the ceramic matrix fired at 1150 °C. b: Details of calcium aluminosilicate. MA: Microgranular aggregates.

appearance of calcium and magnesium aluminosilicates detected by X-ray diffraction. At 1050 °C, the texture remains open (Fig. 5c and d), whereas at temperatures at 1150 °C, the matrix has undergone vitrification in both M1 and M2 mixtures with the result that the clay forms are indistinguishable and the porosity is closed with large circular pores. Some of these pores are connected by "bottlenecks" typical at this temperature (Fig. 5e and f). In both mixtures at temperatures at 1150 °C, there are two distinct phases: the glazed ceramic matrix and the microgranular aggregates of calcium and magnesium aluminosilicate (Figs. 5e, f and 6). These aggregates form rounded surfaces caused by incipient melting and are distributed uniformly throughout the sample in greater number than in those specimens fired at lower temperatures. This result is consistent with the X-ray diffraction analysis.

IGCC slag grains can be clearly distinguished in specimens fired at low temperature (<1100 °C) (Fig. 5b and d) because they are glassy and stand out from the ceramic matrix, which has an open texture. Slag grains retain their original shape and have not started to melt on the surface. The grain boundaries of the slag and their contact with the matrix are very well defined. Above 1100 °C, the ceramic matrix has undergone vitrification and thus, contact between the slag grain boundaries and the matrix is not clearly distinguishable in many cases (Fig. 5f).

Devitrification of the IGCC slag is evidenced by the appearance of incipient crystals on the interior surface of the slag pores, known as "ceramic geodes". These crystals begin to appear at 950 °C (Fig. 7a), and as the temperature increases, they become better defined and more abundant (Fig. 7b and c). The crystal formation

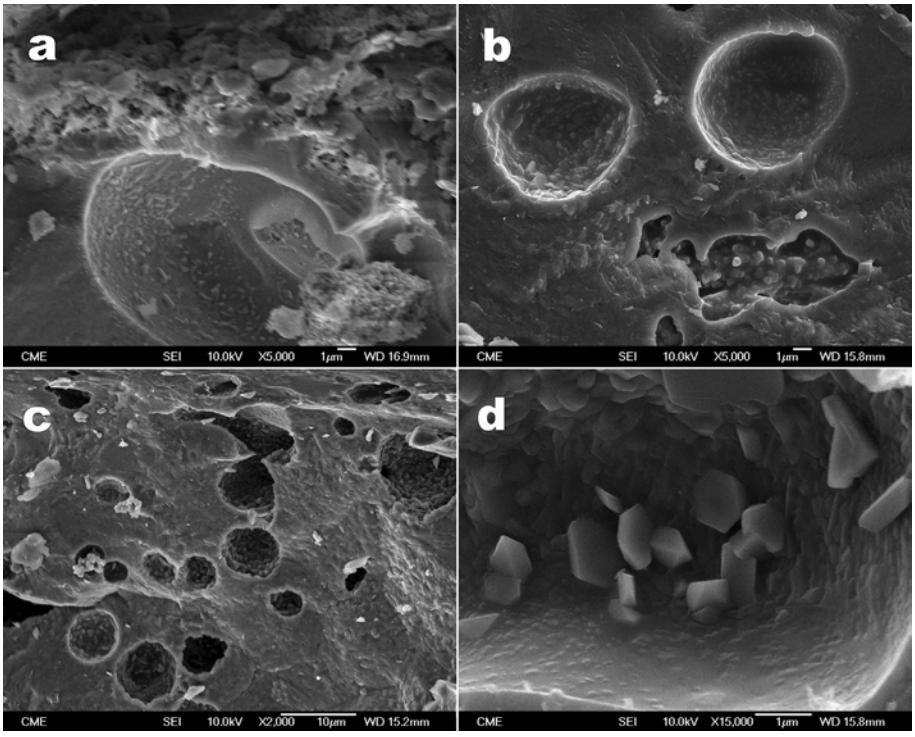

Fig. 7. SEM-FEG micrographs showing devitrification crystals in slag pores. a: 950 °C. b: 1050 °C. c: 1150 °C. d: 1050 °C.

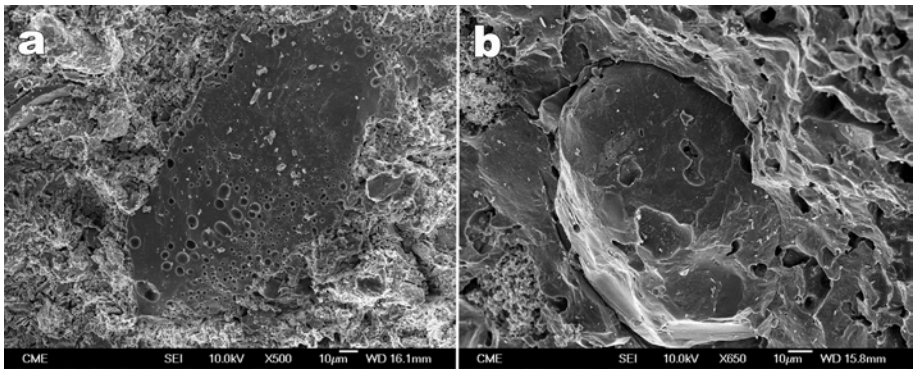

Fig. 8. SEM-FEG micrographs at 1150 °C. a: IGCC slag grain. b: quartz grain.

agrees with the increase of the anorthite peak detected by XRD in ceramic specimens of M2 mixture (Fig. 3c) and also with the anorthite crystals observed by polarized light microscopy in the IGCC slag grains (Fig. 4d and f). These crystals possess different morphologies and sizes. Fig. 4d shows the anorthite crystals.

Devitrification of the IGCC slag explains the better results of the mechanical strength of the ceramic material. When slag grains are heated, their strength increases because they evolve from an amorphous material to a mineralogical material. Usually, the addition of degreasing materials, such as quartz and carbonate sands, decreases shrinkage, which is beneficial to the ceramic mixture. However, these materials have other disadvantages, such as increasing water absorption and decreasing the mechanical strength of ceramics, thereby worsening the properties of the ceramic mixture. The addition of slag as a degreasing material improves the diametral shrinkage and limits water absorption but does not affect the mechanical strength of the material. These results demonstrate that slag is a better degreasing material than either quartz or carbonate sands.

Fig. 8 shows the IGCC slag grains bonded to the ceramic matrix without leaving pores (Fig. 8a), whereas quartz grains are separated from the ceramic matrix (Fig. 8b) as a consequence of a quartz polymorphic transformation at 535 °C. The slag grains and the ceramic matrix are joined (Figs. 5b, d and 8a) due to the thermal behaviour of the IGCC slag (Acosta et al., 2002a). Iron could act as a source of flux in the surface of the grains and favour their cohesion with the ceramic matrix (Figs. 4 and 5).

4. Conclusion

The study performed here shows that IGCC slag, when heated, changes from an amorphous phase to a crystalline but the devitrification process does not affect the shape of the slag grains, which maintain their shape below 1150 °C. There is no reaction between the components of the slag and the ceramic matrix that could lead to new mineral phases. Thus, the mineralogy of the IGCC slag grains and the ceramic matrix evolves as two independent phases.

Furthermore, devitrification within the slag grain results in a more resistant material. By contrast, other degreasing materials either decrease the mechanical strength when heated or decompose, as with carbonate sands. Thus, the addition of IGCC slag does not worsen the mechanical strength of the ceramic material, but, rather, improves its properties.

Because of its physico-chemical and thermal properties, IGCC slag grains do not separate from the ceramic matrix as much as the grains of other degreasing materials. Above 950 °C, there is a degree of softening on the surface of the slag grains, but it does not affect the shape of the grains. This softening leads to good contact between the matrix and the grain boundaries, thereby decreasing the porosity of the material and improving its cohesion. Thus, the slag neither increases water absorption nor decreases mechanical strength.

Analysis of SEM-FEG reveals that the quartz grains are separated from the ceramic matrix, thereby significantly increasing porosity. The increase in porosity explains the increase in water absorption and the decrease in the mechanical strength. However, this phenomenon does not occur with the grains of slag, which are welded to the ceramic matrix. Therefore, IGCC slag is a better degreasing than either quartz or carbonate sands. This superior behaviour is due to the softening temperature of the slag. Thus, IGCC slag used as a degreaser reduces water absorption. This is very important from an industrial standpoint because IGCC slag not only offers an alternative to traditional degreasing agents in the building ceramic industry but also transforms waste into a secondary raw material, thus avoiding its disposal.

The IGCC slag grains are welded to the ceramic matrix. In summary, industrial wastes are made inert, and the properties of ceramics are improved.

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